# LONG-TERM ELECTROCHEMICAL OXIDATION STUDIES OF MULTI-COMPONENT HYDROCARBON FUELS

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## Introduction

For the most part, studies of the various aspects of direct hydrocarbon oxidation are based on short-term measurements. It is recognized that long-term anode performance with hydrocarbon fuels may be different from what can be predicted from short-term polarization data. This is due to such time-dependent factors as the accumulation of inert ad-layers on the electrode surface.* A study of the long term effects that various "unreactive" additives (e.g. aromatics, olefins, naphthenes) may have on the performance of fuel cells operating on n-octane was made in order to determine whether there may be cumulative inhibition of the active sites on the anode as a function of time by these additives.

Other long-term effects of various additives in multi-component, octane-based fuels, are changes in the characteristics of performance cycling, which is present when hydrocarbons are oxidized directly with phosphoric acid electrolytes.

It is the purpose of this study to provide information and shed some light on these time-dependent aspects of anode performance.

## Experimental

The life testing installation has been described earlier (1). The electrodes (both anode and cathode) were of the same types described elsewhere (2) (35 mgms $Pt/cm^2$, 85% Pt-15% TFE, gold-coated Ta screen for anodes, Pt screen for cathodes).

The following experimental procedure was used: After establishing the desired gas flow rates (in general: 20 $\mu\ell$ fuel/min; corresponding to 10 stoichs of octane at 1 amp and 10 stoichs of oxygen supplied in the air stream to the cathode) and isothermal conditions (350°F), the open-circuit potential was recorded. Thereafter, an initial polarization curve was taken.

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* Another important contribution to such performance changes is a progressive structural deterioration of the electrode; particularly, where an optimization of the electrode structure is pursued.

After these initial data were obtained, the cell current was maintained in general at 1.5 amp (30 ASF), by means of Electro-Products, Model EFB, D.C. power supply units. The IR-included anode vs $H_2/H^+$ potential and cell current were continuously recorded with Varian two-channel strip chart recorders. IR-free potential data were also obtained by means of a Kordesh-Marko bridge. These data were logged, together with cell potentials and resistances.

The circulating electrolyte concentration was maintained at 95-98% $H_3PO_4$ by means of controlled-rate addition of water to the electrolyte sump.

At the end of each test (usually caused by excessive electrolyte leakage through the anode) a final polarization curve was taken. The cell was then disassembled and the anode was cleaned. If desired, its surface area was measured by the B.E.T. method.*

Whenever necessary, the polarization curve data points were determined after suitably pre-activating the anodes at anode potentials of > 0.90 v vs $H_2/H^+$, and subsequently waiting for the output to stabilize at what would have been the average anode potential under cycling conditions for a fixed current density. This wait period was usually of the order of three to four minutes. In this way, cycling disturbances were avoided, without affecting the values to be measured.

## Interpretation of the Test Data

### A. Characteristics of Extended Performance - vs - Time Data

As mentioned above, direct hydrocarbon oxidation in phosphoric acid is characterized by spontaneous performance cycling. One of the objectives of the present work was to quantitatively define differences in the behavior of octane and various octane-based binary and multi-component fuel mixtures, in terms of the frequency and/or the amplitdue of anode potential fluctuations at constant current. The interpretation of such differences can be refined by noting that in each case the time required for the onset of cycling can be divided into four distinctly different periods. Figure 1 is a graphical representation of these four periods which can be defined as follows:

I. The induction period - a short interval of time, normally one to two hours, during which there is a large increase in anode potential, from its open-circuit value.

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* Whenever time perimitted, initial surface area measurements on anodes prior to assembly were also made. The method itself, as applied to whole electrodes, has been described earlier.

Technical Summary Report No. 7, Hydrocarbon-Air Fuel Cells, January 1965 - 30 June 1965; ARPA Order No. 247, Contract Nos. DA-44-009-ENG-4909, DA-44-009-AMC-479(T), and DA-44-009-ENG-4853, p. 193 ff.

ibid, No. 8 same contract numbers.

II. The ripple period - follows the induction period and is of short duration; up to two hours. During this time interval, the anode potential starts fluctuating at high frequencies (e.g. several times a minute) and low amplitudes (e.g. 10-20 mv).

III. The onset-of-cycling period - sometimes indistinguishable from the actual cycling mode of operation (IV, below). When it occurs, it lasts a few hours, or less, and is characterized by performance cycling at frequencies that are sometimes higher and sometimes lower than during the actual cycling operation that follows; the corresponding amplitudes are always smaller (e.g. 20-80% of actual cycling amplitudes).

IV. The cycling period - characterizes the onset of steady-state anode operation. Here, the performance fluctuations have been established to their full extent, according to the experimental conditions and the species being oxidized.

The anode potential will change from its minimum to its maximum value according to a set pattern. This pattern can be used to define differences in the behavior of various fuels of interest in terms of frequency and amplitude of performance cycling (see following section: Results and Discussion).

As time goes on, various modes of degradation occur (e.g. progressive catalyst inhibition by a relatively non-reactive ad-layer, or structural degradation of the anode). These may be responsible for observable changes in the characteristics of performance cycling. It sometimes appears that these changes are auto-compensatory in nature; thus, a certain increase in frequency is associated with a decrease in amplitude and vice-versa. This latter observation, however, is only of a tentative nature at our present state of knowledge.

It should be noted that this report summarizes cycling differences, between various species, in terms of the characteristics of period IV and the combined periods (I + II + III) as defined above. Certainly further elaboration is possible, whenever warranted, in terms of individual differences in the characteristics of periods I, II, and III.

B. Processing and Significance of the Experimental Data

The high-loading unsupported platinum anodes used in the present work, while sufficiently active catalytically to yield meaningful results, at least on a comparative basis, present the drawback of structural variance with time. This variance is sufficiently severe to make it necessary not to consider the "last-stage" data for comparison purposes since they are often influenced by high rates of electrolyte leakage through the anode.

Two methods of data interpretation were used:

1) Use of available polarization curve data to define various performance points (e.g. anode potential at given current densities) at various chronological

stages; say, at the beginning and at the end* of a run. These are, essentially, extent-of-degradation data.

2) Averaging of cycling data, from recorder traces, and graphical representation of the resulting changes of performance with time. These are the typical life test curves, supplying information on the total life for a set of predetermined conditions, as well as the individual durations of the various periods of operation (induction, ripple, etc.).

## Results and Discussion

### A. n-Octane

Seven anodes were tested with pure n-octane in order to establish a reliable frame of reference for the results obtained with various fuel mixtures. Table 1 lists a variety of averaged performance data for n-octane; the ranges of values from which the averages were calculated are given in parentheses. The broad band in Figure 2 is a graphical representation of the change in anode potential with time for six of the seven anodes, based on an averaging of the cycling performance as described in the experimental section. The boundaries of the band represent the range of anode performance for the electrodes tested. The abrupt break in the curve after the initial several hours is an indication of the onset of steady-state operation (cycling).

Table 2 summarizes the cycling characteristics observed with n-octane. The numbers marked in parentheses in the fourth column represent the time intervals during which the reported frequencies were observed. The range of frequency values are arranged chronologically with the left-hand figures representing the beginning of the measured period and the right-hand figures, the end. Some resistive load operational data is also included for comparison purposes.

The relationship between current density and time-to-cycle, at constant-current conditions, follows an exponential behavior previously described (2) with the log (time to cycle) decreasing as the current density increases.

The frequency of cycling appears to indicate a trend towards higher values at higher current densities while the amplitude does not exhibit any significant trend. It is interesting to note that, under resistive load conditions, the amplitude does not appear to be different from that at constant current (common basis of comparison: 30 ASF), although the current varies as well. The averaged anode potential is higher, though, indicating that simultaneous current and voltage cycling might have a deleterious effect on performance.

Regarding the time-to-cycle values (Column 3 in Table 2), equivalent to periods I + II + III, it is interesting to note that the ripple period (II)

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* Note that the end-of-run data are not to be confused with the previously mentioned "last-stage" data; the end-of-run data are obtained before the electrode degrades to a point where high rates of electrolyte leakage can occur.

Table 1

Average Life Test Data for n-Octane Oxidation at 350°F

(Ranges of values corresponding to the averages are marked in parentheses.)

Electrolyte: 95-98% $H_3PO_4$ Anode: 35 mgms Pt/$cm^2$
Life: 190 hours (150-405)

| | |
|---|---|
| Cause of test termination: | Excessive anode leakage |
| Initial anode resistance: | 6 mΩ (0.004-0.008) |
| Initial IR-included anode potential at 30 ASF: | 0.436 v (0.380-0.470) |
| Final IR-included anode potential at 30 ASF: | 0.504 v (0.455-0.600) |
| Rate of decay of potential: | approx. 0.45 mv/hr (0.100-0.870) |
| Initial IR-included power output at 30 ASF: | 12.7 WSF (11.4-13.5) |
| Initial IR-included power output at 60 ASF: | 18.4 WSF (15.6-20.4) |
| Final IR-included power output at 30 ASF*: | 11.2 WSF (8.7-11.4) |
| Final IR-included power output at 60 ASF*: | 13.3 WSF (7.8-15.6) |

*The pre-activation procedure described in the Experimental Section was used prior to obtaining these data.

included therein was at times negligibly small, while at other times it contributed significant portions to the total reported time interval (e.g. 2-3 hours at 30 ASF). The onset-of-cycling period (III) was often indistinguishable from the actual cycling period (IV).

## B. Normal Octane with Single Additives

A comparison of the life test summaries in Tables 1 and 3 indicates that there are no significant performance differences in the cycling period for the octane + 1% aromatic and octane + 3% olefin data as compared to pure octane. These similarities can be seen more clearly in Figure 2 where the performance-time curves for the fuels containing olefins and aromatics fall within the n-octane band. It can also be seen that anodes operating on octane + 5% naphthene fuels polarize more extensively during a relatively short initial time interval. Even after this brief interval, the performance-time curve is situated within the upper portion of the n-octane band while the latter has most of its data clustered in its lower portion.

The cycling characteristics of octane plus single additives are summarized in Table 4. The only significant differences, compared to pure octane oxidation, are the increased cycle frequencies, roughly 3 times higher, for the binary fuels.

Table 2

Cycling Characteristics for the Oxidation of n-Octane at 350°F in a 95-98% Phosphoric Acid System

| No. of Anodes Represented | Current Density, ASF | Time-to-cycle,* hours | Cycle Frequency, CPH | Amplitude (potential range), mv vs $H_2/H^+$ at anode | Average Anode potential vs $H_2/H^+$, mv |
|---|---|---|---|---|---|
| 2 - | 20 | 7.0 | 7-3 (24 hrs) | 300-720 | 530 |
| 5 - | 30 | 4.0 | 4-3 (59 hrs**) | 363-750** | 560 |
| 1 - | 60 | 1.2 | 8-12 (4 hrs) | 300-720 | 560 |
| 1 - | 100 | 0.25 | 40-28 (4 hrs) | 370-800 | 600 |
| Note: the figures below represent some data collected for a resistive load mode of operation (cell #LT 142) with both current and voltage cycling. | | | | | |
| 1 - | 2 (1.8-2.4) | 5 | 12-20 (35 hrs) | 450-680 | 500 |
| 1 - | 6 (5.8-6.2) | 4 | 20-6 (24 hrs) | 400-680 | 570 |
| 1 - | 30 (28-33) | 5 | 10 | 360-750 | 600 |

* Corresponding to periods I + II + III (see interpretation of the test data).

** Most of the time at 3 CPH; the amplitude for 4 CPH: 385-680 mv.

Table 3

Life Test Data for the Oxidation of n-Octane with Single Additives at 350°F in a 95-98% Phosphoric Acid System

Anode: 35 mgms Pt/cm$^2$

| Cell No. | Mole % Fuel Additive to n-octane | Time, hours | C.D.,* ASF | P.D.,* WSF | Life,** hours | Anode* Res., ohms | Anode/$H_2$ Ref Potential, volts |
|---|---|---|---|---|---|---|---|
| | Olefins | | | | | | |
| 145 | 3% 2-Methyl butene-1 | 1 | 30 | 14.4 | 80 | 0.009 | 0.400 |
| | | | 60 | 21.0 | | | 0.480 |
| | | 79 | 30 | 12.9 | | | 0.430 |
| | | | 60 | 15.7 | | | 0.505 |
| 146 | 3% Octene-2 | 1 | 30 | 13.8 | 391 | .008 | 0.440 |
| | | | 60 | 19.8 | | | 0.510 |
| | | 382 | 30 | 12.9 | | | 0.410 |
| | | | 60 | 17.4 | | | 0.490 |
| 151 | 3% 2-Methyl butene-1 | 1 | 30 | 12.6 | 215 | .007 | 0.440 |
| | | | 60 | 15.6 | | | 0.460 |
| | | 215 | 30 | 11.1 | | | 0.455 |
| | | | 60 | 12.9 | | | 0.540 |
| | Aromatics | | | | | | |
| 147 | 1% 1, 2, 4 Trimethyl-benzene | 1 | 30 | 12.6 | 94 | .003 | 0.440 |
| | | | 60 | 12.0 | | | 0.610 |
| | | 55 | 30 | 9.3 | | | 0.640 |
| | | | 60 | 0.0 | | | +1.00 |
| 149 | 1% Toluene | 1 | 30 | 11.4 | 387 | .006 | 0.455 |
| | | | 60 | 16.8 | | | 0.480 |
| | | 387 | 30 | 9.1 | | | 0.500 |
| | | | 60 | 10.5 | | | 0.580 |
| 157 | 1% 1, 2, 4 Trimethyl-benzene | 2 | 30 | 13.2 | 242 | .007 | 0.395 |
| | | | 60 | 19.8 | | | 0.440 |
| | | 235 | 30 | 11.7 | | | 0.470 |
| | | | 60 | 18.0 | | | 0.490 |
| | Naphthenes | | | | | | |
| 143 | 5% Methyl-cyclohexane | 1 | 30 | 12.5 | | .003 | 0.440 |
| | | | 60 | 18.0 | 144 | | 0.500 |
| | | 33 | 30 | 9.0 | | | 0.540 |
| | | | 60 | 0.0 | | | +1.000 |
| 156 | 5% Methyl-cyclohexane | 1 | 30 | 12.6 | 195 | .008 | 0.465 |
| | | | 60 | 18.6 | | | 0.510 |
| | n-octane | 194 | 30 | 9.8 | | | 0.555 |
| | | | 60 | 12.0 | | | 0.635 |

* Data from polarization curves.

** Actual duration of test before excessive anode leakage required shut-down of the cell.

Table 4

Cycling Characteristics for n-Octane with Single Additives at 30 ASF at 350°F in a 95-98% Phosphoric Acid System

Anode: 35 mgms Pt/$cm^2$

| Fuel | Time-to-Cycle hours | f/(Amplitude)/time CPH/(mv)/hours | Avg. Anode Potential (IR-included), mv vs $H_2/H^+$ |
|---|---|---|---|
| Olefin Additives | | | |
| 3 m/o 2-methylbutene-1 | 4.0 | 24/(250-710)/22 | 505 |
| 3 m/o 2-methylbutene-1 | 3.0 | 5/(330-730)/23 | 560 |
| 3 m/o 2-methylbutene-1 | 4.5 | 5/(340-730)/75 | 560 |
| Aromatic Additives | | | |
| 1 m/o Toluene | 4.0 | 5/(400-750)/25 | 550 |
| 1 m/o 1, 2, 4 Trimethyl-benzene | 4.5 | 6/(360-750)/28 | 590 |
| Naphthene Additives | | | |
| 5 m/o Methylcyclohexane | 4.0 | 12/(350-720)/15 | 580 |
| 5 m/o Methylcyclohexane | 0.6 | 9/(300-690)/24 | 580 |

C. Normal Octane with Several Additives

It has been shown (3) that there is no cumulative effect for multiple additions of "unreactives" up to a total of 11 mole % additives. It was shown, in fact, that the performance penalty roughly corresponded to the "worse offender."

A summary of the life-test data for fuels containing several additives is given in Table 5. The fuels are listed in order of increasing concentration of additives.

Figure 3 represents performance-time data for four cells operated on mixtures of 50/50 normal and iso-octane* containing 11 mole % additives (1% aromatic, 5% naphthene, 5% olefin). It is seen that there is first a sharp increase in anode potential to a value of about 0.575 volt where the potential finally stabilizes. Comparison of this curve with the octane data given in Figure 2 indicates that the multi-component fuel data fit within the upper portion of the n-octane band. The approximately 40 mv penalty over the performance of pure n-octane, based on the 5% naphthenes being the "worse offender" under these conditions is substantiated by these graphical representations. This is due to the fact that most of the n-octane results fall within the lower portion of the performance-time band while the multi-component data are clustered close to the upper limit.

The situation appears to become different for higher amounts of additives. Two cells operating on 50/50 n + i-octane containing 26% additives (1% aromatic, 5% olefins, 5% cyclohexanes, 15% cyclopentanes) were life tested. The variation of the anode potential with time is shown in Figure 4. When the data given in this figure is compared with the octane data in Figure 1 it appears that there is at least a 40 mv anode potential increase above the upper limit of the octane data band (i.e. 40 mv higher than what the "worse offender" would normally show). This performance difference could be due either to the presence of methylcyclopentane or to the high total amount of additives. It is expected that these aspects will be clarified in future work.

The balance of Table 5 is a summary of the life test data where increasing amounts of the "unreactive" components were added to the fuel. The data is shown diagramatically in Figure 4 and 5. Since these concentrations of "unreactives" were not studied on a short term basis the discussion of these results will be deferred until this data is on hand. However, it is evident that sizeable performance penalties are paid when the unreactive concentrations reach such high levels (greater than 11%). It can be seen that when successive amounts of "unreactive" compounds are added the performance penalty increases until the deviation from n-octane reaches about 180 mv** (~50% of the cell output IR included). This performance drop occurs within the first 25 hours of operation, where effects of anode deterioration are minimal. Figure 6 is a graphic representation of the

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* It has been previously shown (3) that there are no performance differences between anodes operating on n-octane and on n-octane + i-octane mixtures.

** Note that the detrimental effect of octene-1, as compared to octene-2 particularly obvious with high amounts of additives (30 mole %).

Table 5

Multiple Addition Performance of a 50/50 n + iso-Octane Based Fuel at 350°F in 95-98% Phosphoric Acid System

Anode: 35 mgms Pt/cm$^2$

| Fuel | Time, hours | C.D.* ASF | P.D.* WSF | Life,** hours | Anode/$H_2$ Ref Potential, volts | Anode Res., ohms |
|---|---|---|---|---|---|---|
| 11% Additive (4 cells) | 1 | 30 | 11.5 | 207 | 0.474 | 0.007 |
| | | 60 | 15.3 | | 0.533 | |
| 1 Toluene, 5 Octene-2, 5 M.C.H. | 174 | 30 | 3.9 | | 0.750 | |
| | | 60 | 0.0 | | 1.00 + (anode polarized) | |
| 26% Additive (2 cells) | 1 | 30 | 11.6 | 75 | 0.477 | 0.008 |
| | | 60 | 16.3 | | 0.538 | |
| 1 Toluene, 5 octene-2, 5 M.C.H., 15 M.C.P. | 74 | 30 | 0.0 | | 1.00 + (anode polarized) | |
| | | 60 | 0.0 | | 1.00 + (anode polarized) | |
| 30% Additive (2 cells) | 1 | 30 | 10.3 | 165 | 0.495 | 0.010 |
| | | 60 | 12.3 | | 0.560 | |
| 5 Toluene, 5 Octene-2, 5 M.C.H., 15 M.C.P. | (Both cells overtemperatured > 370°F during initial 24-hour test period) | | | | | |
| 30% Additive (1 cell) | | | | | | |
| 5 Toluene, 5 Octene-1, 5 M.C.H., 15 M.C.P. | 1 | 30 | 6.3 | 38 | 0.640 | 0.010 |
| | | 60 | 4.5 | | 0.710 | |
| | 38 | 20 | 4.0 | | 0.680 | |
| | | 30 | 0.0 | | 1.00 + | |
| 36% Additive (2 cells) | 1 | 30 | 7.6 | 103 | 0.628 | 0.008 |
| | | 60 | 6.6 | | 0.708 | |
| 1 Toluene, 5 Octene-2, 5 M.C.H., 25 M.C.P. | 103 | 30 | 0.8 | | 0.900 | |
| | | 60 | 0.0 | | 1.00 + (anode polarized) | |
| 50% Additive (1 cell) | 1 | 30 | 9.0 | 190 | 0.530 | 0.008 |
| | | 60 | 10.3 | | 0.620 | |
| 5 Toluene, 5 Octene-2, 10 M.C.H., 30 M.C.P. | 187 | 10 | 3.2 | | 0.610 | |
| | | 30 | 0.0 | | 1.00 + (anode polarized) | |
| 55% Additive (2 cells) | 1 | 30 | 7.7 | 110 | 0.645 | 0.006 |
| | | 60 | 8.4 | | 0.700 | |
| 5 Toluene, 10 Octene-2, 10 M.C.H., 30 M.C.P. | 110 | 5 | 1.25 | | 0.720 | |
| | | 10 | 0 | | 1.00 + | |
| 35% Additive (1 cell) | 1 | 30 | 7.8 | 95 | 0.585 | 0.006 |
| | | 60 | 10.2 | | 0.610 | |
| 10 Toluene, 5 Octene-2, 5 M.C.H., 15 M.C.P. | 95 | 0 | 0.0 | | 1.00 + (anode polarized) | |

M.C.P. = methylcyclopentane
M.C.H. = methylcyclohexane

* From polarization curves.

** Actual duration of test before excessive anode leakage or performance below 5.0 WSF dictated test termination.

decrease in current density, at a given polarization, with increasing amounts of the "unreactive" components. It should be noted that the current densities are normalized in terms of real B.E.T. surface areas measured on the pure electrodes. The spread of the data increases at higher concentrations of "unreactives."

The cycling characteristics of these multi-component fuels are shown in Table 6. The numbers in parentheses have the same meaning as described for Table 2.

These results were averaged for operation at 30 ASF. The frequency and amplitude of cycling does not appear to exhibit any of the trends reported for n-octane and n-octane with single additive mixtures. In fact, there are no appreciable changes of the frequency of cycling with time.

## Conclusions

Information regarding the influence that various hydrocarbon additives have on the short-term performance of an octane anode, cannot necessarily be applied towards predictions of long-term performance in that any detrimental additive effects noticeable during a short run may become insignificant in the context of real fuel cell operation, proceeding for extended periods of time.

However, while several additives do not necessarily have to affect performance on a cumulative detrimental basis, beyond a certain quantity limit and for extended durations of operation, the overall decrease in anode performance can be intolerably severe.

The cycling characteristics associated with the process of direct electrochemical oxidation of hydrocarbons can be described in terms of several distinct modes of performance fluctuation prior to the establishment of steady-state conditions.

This descriptive refinement can be quite useful as a semi-quantitative tool for the definition of operational differences in the expected oxidation process, as caused by a variety of additives to a base fuel.

## Acknowledgement

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## References

1. Technical Summary Report No. 5, Hydrocarbon-Air Fuel Cells, January 1964-June 1864, ARPA Order No. 247, Contract Nos. DA-44-009-ENG-009-ENG-479(T).

2. ibid., Report No. 5, p. 4-145

3. ibid., Report No. 9, p. 2-107 ff.

Table 6

Cycling Characteristics for n-Octane with Multiple Additives at 30 ASF at 350°F in a 95-98% Phosphoric Acid System

Anode: 35 mgms Pt/$cm^2$

| Fuel | Time-to-Cycle, hours | f(Amplitude) CPH/(volts) | Avg. Anode/$H_2$ Ref, volts |
|---|---|---|---|
| 1 Toluene, 5 octene-2, 5 MCH* | 5 | 15(446-662)-11(415-685) | 0.595 |
| 1 Toluene, 5 octene-2, 5 MCH, 15 MCP | 6 | 19(525-675)-17(500-700) | .640 |
| 1 Toluene, 5 octene-2, 5 MCH, 25 MCP | 1 | 12(485-685)-10(435-720) | .655 |
| 5 Toluene, 5 octene-2, 5 MCH, 15 MCP | 10 | 10(543-720)-12(445-745) | .645 |
| 5 Toluene, 5 octene-2, 10 MCH, 30 MCP | 3 | 11(560-750)- 9(420-820) | .710 |
| 5 Toluene, 10 octene-2, 10 MCH, 30 MCP | 1 | 10(500-720)-15(450-760) | .725 |

*MCH - Methylcyclohexane.

MCP - Methylcyclopentane.

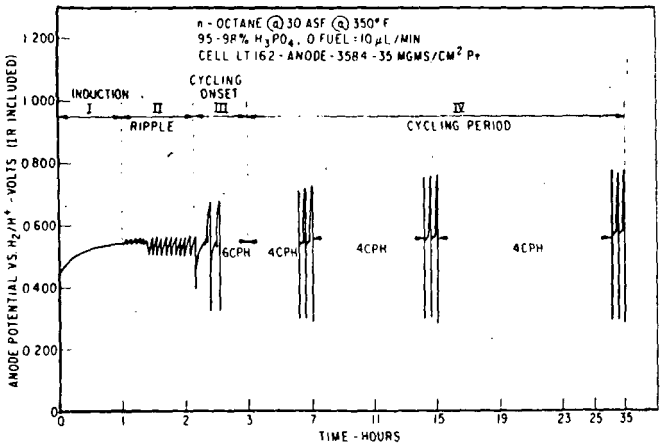


Figure 1 Typical Anode Performance at Constant Current Over Extended Time Periods.

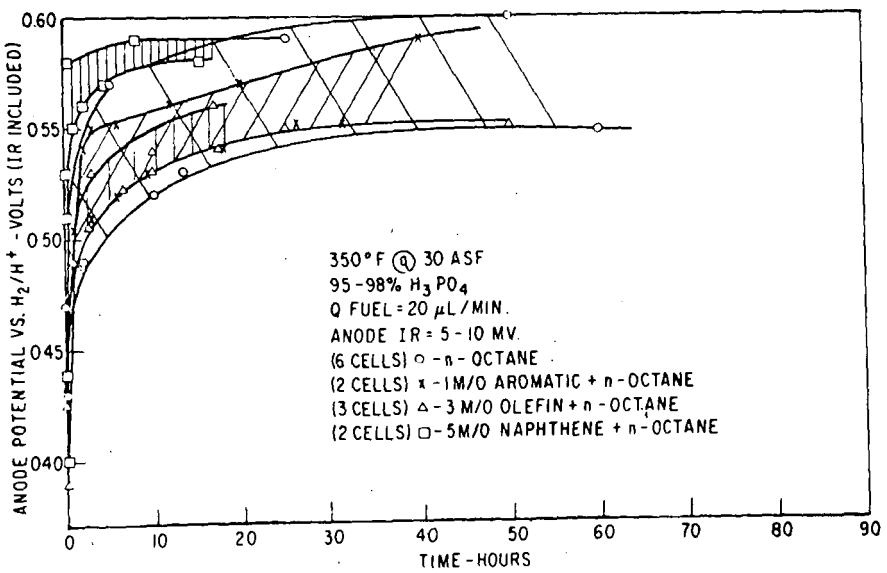


Figure 2 Change of Anode Potential with Time.

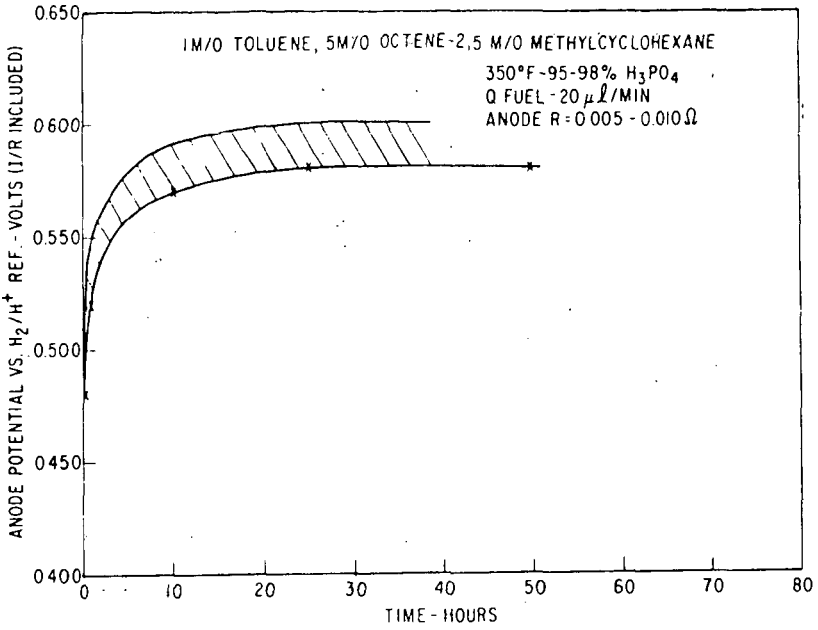


Figure 3 Change of Anode Potential with Time (Unsupported Anode: 35 mgms Pt/cm$^2$).

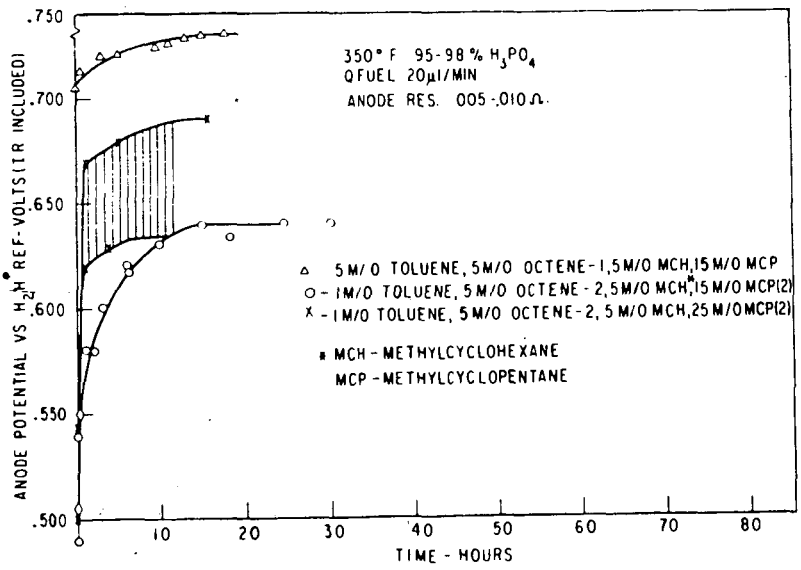


Figure 4 Change of Anode Potential with Time (Unsupported Anode: 35 mgms Pt/cm$^2$).

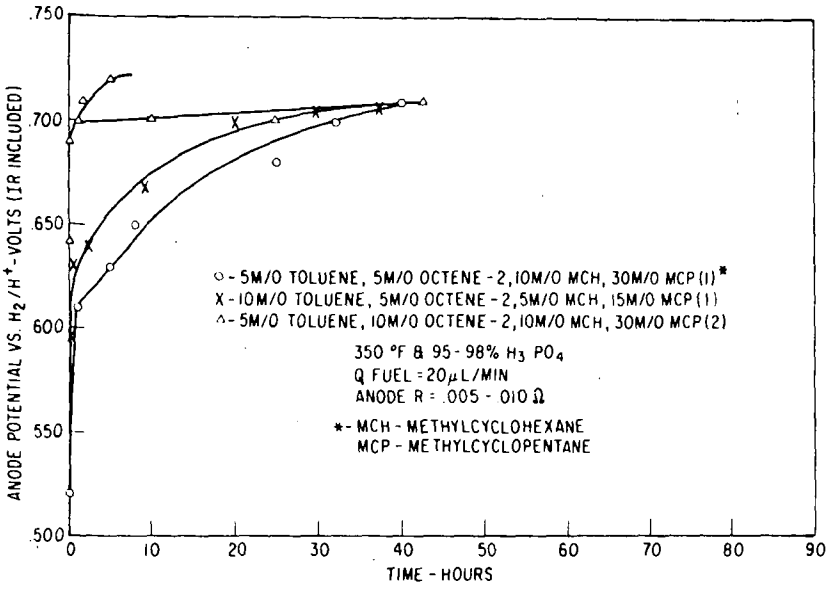


Figure 5 Change of Anode Potential with Time (Unsupported Anode: 35 mgms $Pt/cm^2$).

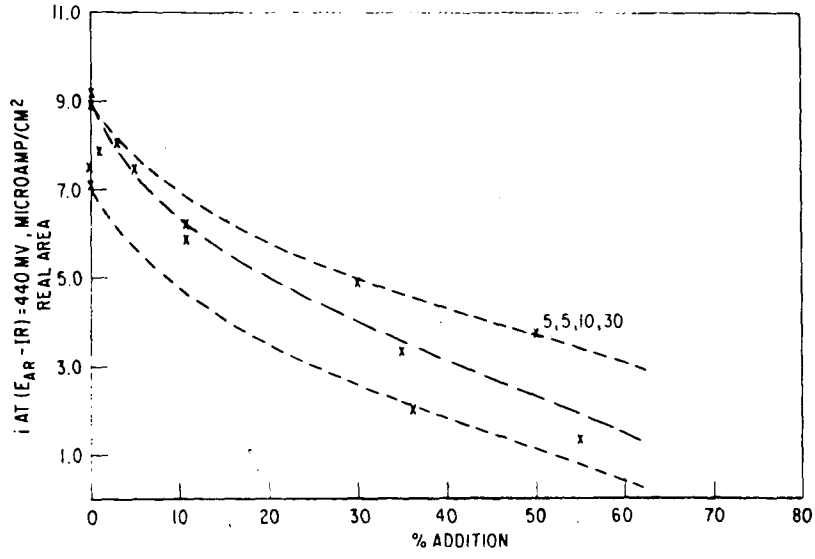


Figure 6 True Current Densities as a Function of the Total Amount of Additives.